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FLAMMABILITY CHARACTERISTICS OF FIBER-REINFORCED EPOXY COMPOSITES FOR COMBAT VEHICLE APPLICATIONS

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POLYMER RESEARCH BRANCH



August 1992

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ABSTRACT

The use of composites in U.S. Army systems as a means of decreasing weight and enhancing survivability, without reducing personnel safety, has been considered for some time. The U.S. Army Materials Technology Laboratory (MTL) successfully demonstrated in an earlier program that a ground vehicle turret could be fabricated from fiber-reinforced composite material. This technology was successfully extended to the fabrication of a composite vehicle hull in an earlier phase of the current program.

Organic polymers are one of the major constituents of fiber-reinforced composites. As components of military systems these materials are expected to survive combustion and pyrolysis processes associated with fires. It is, therefore, necessary to develop an understanding of the flammability behavior of composite materials in the early design stages of a military vehicle such as the Composite Infantry Fighting Vehicle (CIFV), the Advanced Systems Modification (ASM), or any future U.S. Army combat vehicle.

The present study attempts to characterize the flammability behavior of composite materials associated with Phase III of the CIFV Hull Program in terms of accepted fire-resistant material evaluation parameters.

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INTRODUCTION

Fiber-reinforced composite materials are used extensively because of their physicochemical properties and their high strength/weight ratio. The use of composites in U.S. Army systems as a means of decreasing weight and enhancing survivability, without reducing personnel safety, has been considered for some time. The U.S. Army Materials Technology Laboratory (MTL) has successfully demonstrated in an earlier program¹ that a ground vehicle turret could be fabricated from fiber-reinforced composite material. That technology was subsequently applied to the fabrication of a composite hull for the Composite Infantry Fighting Vehicle (CIFV).^{2,3}

Organic polymers are one of the major constituents of fiber-reinforced composites. As components of military systems these materials are expected to survive combustion and pyrolysis processes associated with fires. It is, therefore, necessary to develop data, and an understanding of the flammability behavior of composite materials, in the early design stages of a military vehicle such that assessments can be made of potential hazards and the type of protection that may be required. This report describes the results of a study on fiber-reinforced epoxy composite materials (see Table 1) which was undertaken to quantify data and gain an understanding of the processes associated with combustion, pyrolysis, fire propagation, and fire extinguishment.

Table 1. CANDIDATE COMPOSITE MATERIALS

Sample No.	Fiber/Resin	Ratio	Comments
MTL #6	S2/Epoxy	65/35	Ferro Corp. CE-321R
MTL #7	S2/Epoxy	65/35	ICI-Fiberite MXB 7701
MTI #8	S2/Epoxy	65/35	American Cyanamide CYCOM 5920 (X920)

NOTE: Fiber-reinforced composites for Phase II, MTL #1 through #5, were characterized and reported earlier.^{4,5}

In the study, laboratory scale techniques were used to quantify the following: thermal response by thermal analysis techniques, ease of ignition by oxygen index and its dependency on temperature, smoke generation by smoke density (SD) measurement, and pyrolysis effluent composition by pyrolysis-gas chromatography/mass spectrometry (GC/MS). Simultaneously, an independent evaluation of the same composite materials was initiated by Factory Mutual Research Corporation (FMRC), under contract to MTL, to further define the flammability characteristics of fiber-reinforced composite materials by laboratory methods unique to that organization.

At FMRC the composite material test specimens will be evaluated in terms of critical heat flux (the minimum heat flux at or below which there is no ignition), thermal response of the material expressed in terms of ignition temperature, thermal conductivity, density, specific heat, heat of gasification, chemical heat of combustion and its convective and radiative components, fire propagation rate, yields of various chemical compounds (e.g., CO, CO₂, total gaseous hydrocarbon, soot particulates), and optical properties of smoke, flame radiative heat flux (expected in large scale fires), and flame extinction using Halon 1301. The results of that evaluation will be reported by FMRC in a technical report prepared for MTL at the conclusion of the contract.

1. SULLIVAN, F. R. *Reinforced Plastic Turret for M2/M3*. FMC Corporation Final Report, Contract DAAG46-83-C-0041, U. S. Army Materials Technology Laboratory, MTL TR 87-39, August 1987.
2. WEERTH, D. E. *Composite Infantry Fighting Vehicle (CIFV) Program - Phase I*. FMC Corporation Interim Report, Contract DAAL04-86-C-0079, U.S. Army Materials Technology Laboratory, MTL TR 89-23, March 1989.
3. PARA, P. R. *Composite Infantry Fighting Vehicle (CIFV) Program - Phase II*. FMC Corporation Interim Report, Contract DAAL04-86-C-0079, U.S. Army Materials Technology Laboratory, MTL TR 91-34, September 1991.
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5. TEWARSON, A. *Characteristics of Fiber-Reinforced Composite Materials*. Factory Mutual Research Corporation, TR JIOP2N1.RC070(A), June 1990.

EXPERIMENTAL

Thermal Analysis (TGA, DTG, ID)

A thermogravimetric analysis (TGA) system, consisting of a TA Instrument 9900, computer controlled, thermal analyzer, and 951 TGA module was employed to determine sample mass loss as a function of temperature in a flowing gas atmosphere (air or helium) as appropriate at a preset flow rate of 100 cc/min. Experiments were conducted in both the dynamic and isothermal mode. The resultant data indicates the thermal stability of the material being examined. In general, materials that are thermally stable are less flammable than those that are thermally labile, since the concentration of low molecular weight combustible fragments is decreased at any given temperature up to the point where major decomposition of the material occurs.

The isothermal decomposition experiment is a variation of the dynamic TGA measurement described above. The same apparatus is employed with the same atmosphere existing within the apparatus; however, in this experiment the thermal level is fixed and the change in sample mass is recorded as a function of time. The experimental results indicate the ability of the test specimen to withstand a sudden exposure to elevated temperatures at preset levels.

Oxygen Index (OI)

As a measure of susceptibility to ignition, values of OI were determined for the composite material specimens employing a Stanton-Redcroft FTA Oxygen Index apparatus. Specimens were evaluated according to the provisions of ASTM D 2863. The results indicate the minimum concentration of oxygen that is required by the material being examined to sustain equilibrium combustion. Materials with low oxygen indices (21% or less) can be expected to burn readily in normal atmospheric conditions. Materials with moderate oxygen indices (from 21% to 27%) may be expected to ignite, with some increasing difficulty, and to self-extinguish upon removal of the flame source or in normal atmospheric oxygen concentration.

Temperature Dependence of OI

To evaluate the change in OI as a function of temperature, a series of experiments for each material was conducted with a Stanton-Redcroft HFTA apparatus. With this system it was possible to repeat the OI determination with the test specimen at temperatures between ambient and 300°C. The results indicate the change in oxygen requirements to sustain combustion of the sample as the exposure temperature is allowed to increase. By determining the OI at several temperature levels it is possible to plot a profile of the change in ignition behavior of the material as OI versus T.

Smoke Density (SD) Measurements

To determine the SD values for each material, measurement of smoke generation was conducted in an NBS Smoke Chamber. Specimens were evaluated in smoldering and flaming modes according to the provisions of ASTM E 662. In the smoldering mode the test specimen is subjected to the thermal energy of a precalibrated electric heating element adjusted such that the sample surface receives 2.5 watt/sq. cm, at which level the surface temperature is approximately 350°C. In the flaming mode the smoldering conditions are augmented with a six-jet propane burner oriented to impinge flame on the lower portion of the test specimen. The SD value is determined by the decrease in light transmission as measured by a photometer. Values of optical density are quoted, as appropriate, with larger values indicating more smoke produced by the sample being tested.

Pyrolysis-Gas Chromatography/Mass Spectrometry (GC/MS)

To evaluate pyrolysis effluent composition samples of approximately 2 mg mass were pyrolyzed in flowing helium using a Chemical Data Systems (CDS) platinum coil pyrolysis probe, set at 900°C, controlled by a CDS Model 122 Pyroprobe in normal mode. Effluent components were separated on a 12 meter fused capillary column with a cross-linked 5% phenyl, 95% methylsiloxane stationary phase. The GC column was temperature programmed from -50°C to 350°C. Component identification was accomplished with a Hewlett-Packard Model 5995C low resolution quadrupole GC/MS system. Data acquisition and reduction was accomplished using a Hewlett-Packard Model 1000 E-series computer running revision E RTE-6/VM software.

RESULTS

Thermal Analysis

Data representative of the dynamic thermogravimetric analysis experiments are presented in Table 2. Graphic plots of mass loss as a function of temperature are shown in Figure 1. Presentation in this format permits direct comparison of experimental results.

Table 2. THERMOGRAVIMETRIC ANALYSIS RESULTS

Sample No.	Temperature (°C)	Event/Condition	Wt. (%)
MTL #6	Amb - 200	Steady State	0.0
	200 - 500	Mass Loss	21.5
	500 - 800	Mass Loss	14.5
	900	Steady State	64.0
MTL #7	Amb - 200	Mass Loss	0.5
	200 - 500	Mass Loss	25.5
	500 - 800	Mass Loss	12.0
	900	Steady State	62.0
MTL #8	Amb - 200	Steady State	0.0
	200 - 500	Mass Loss	21.0
	500 - 800	Mass Loss	10.0
	900	Steady State	69.0

Data representative of the results of isothermal decomposition experiments are presented in Table 3. A graphic plot of mass loss as a function of time for one of the composite specimens, MTL #7, is shown in Figure 2.

Table 3. ISOTHERMAL DECOMPOSITION PERCENT MASS LOSS DURING FIVE MINUTE EXPOSURE

Sample No.	300°C	400°C	500°C	% Residue 500°C
MTL #6	1.0	12.0	29.5	70.5
MTL #7	3.0	19.0	27.0	73.0
MTL #8	1.0	21.0	28.0	72.0

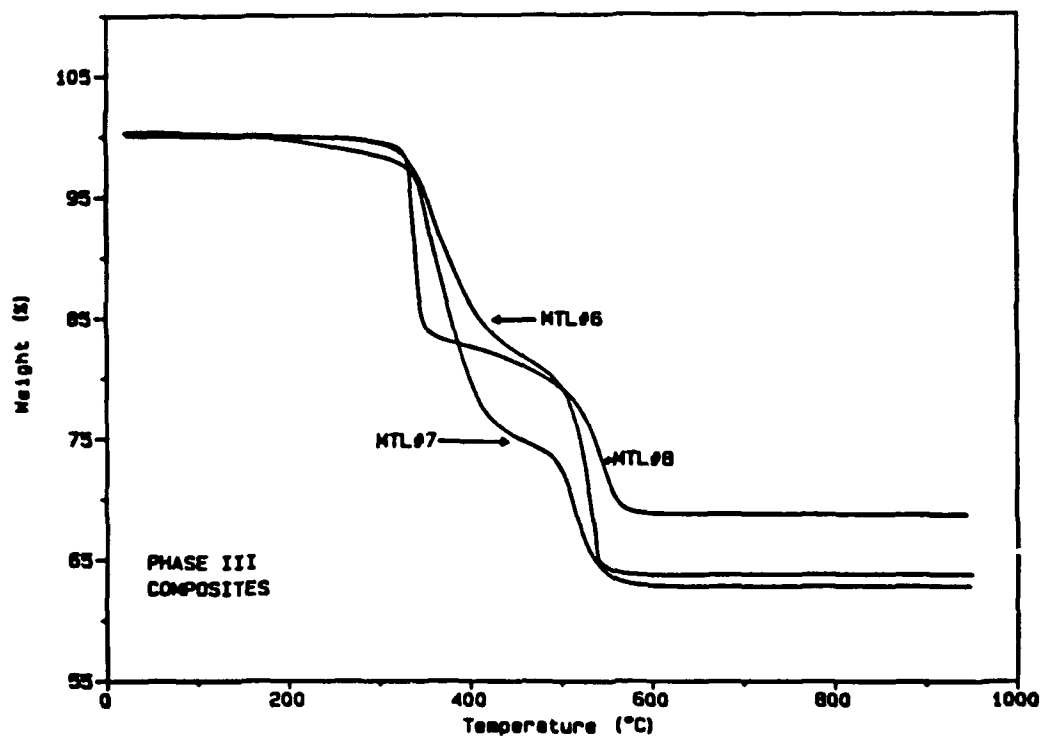


Figure 1. Dynamic thermogravimetric analysis, MTL #6, #7, and #8.

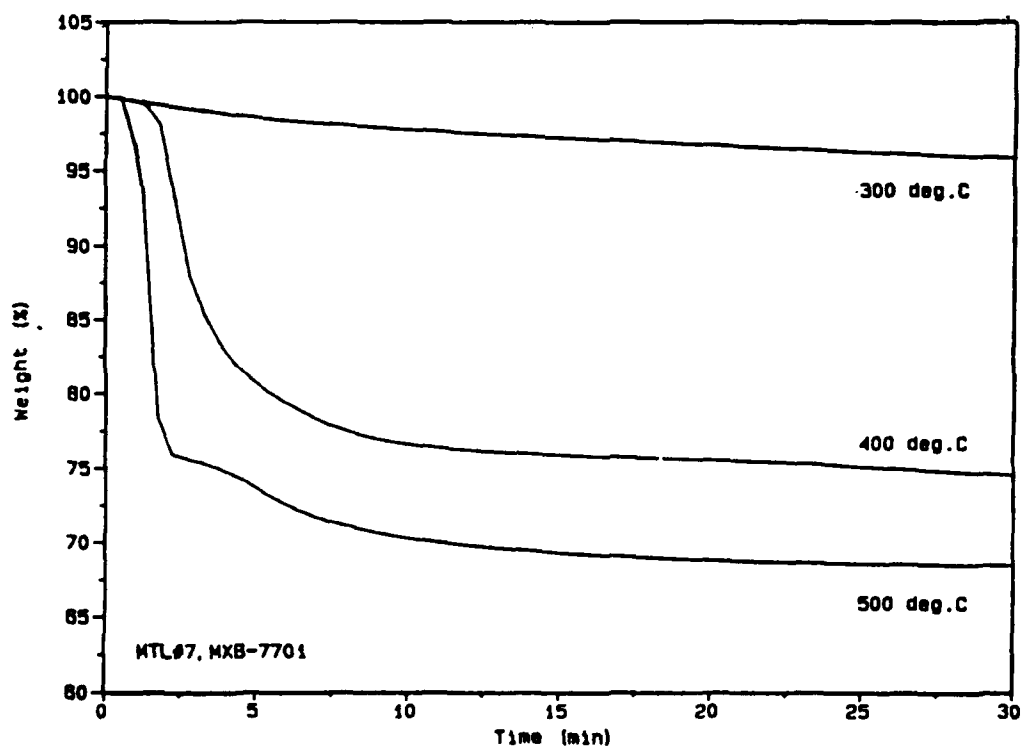


Figure 2. Isothermal decomposition of glass fiber-reinforced epoxy resin MTL #7.

Oxygen Index (OI)/Temperature Dependence of OI

The results of experimental determination of the OI and the temperature dependence of OI are presented in Table 4. Graphic plots are shown in Figure 3.

Table 4. THE OXYGEN INDEX AND TEMPERATURE DEPENDENCE OF OXYGEN INDEX

Temperature (°C)	MTL #6	MTL #7	MTL #8
25	38	50	43
100	43 - 44	59 - 60	54 - 55
200	34 - 35	49 - 50	47 - 48
300	17 - 18	24 - 25	27 - 28

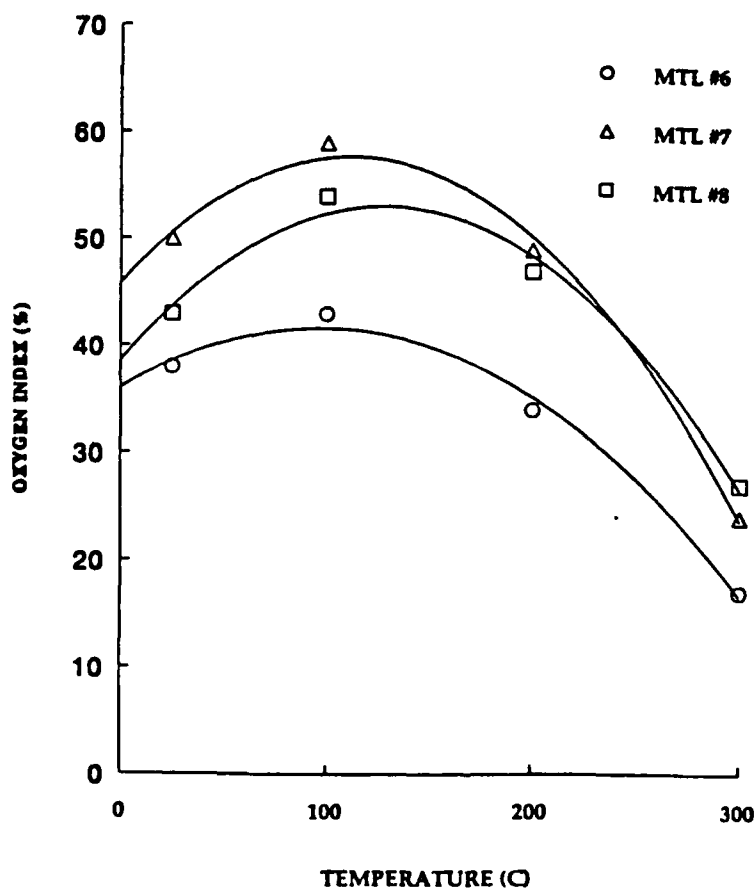


Figure 3. Temperature dependence of OI, MTL #6, #7, and #8.

Smoke Density (SD) Measurement

Table 5 contains the results of SD measurements made with samples of MTL #6 through #8. A representative graphic plot of smoke generation as a function of decreasing light transmission is shown in Figure 4.

Table 5. SMOKE DENSITY OF S2/EPOXY COMPOSITES

	Smoldering	Flaming
MTI #6 - FERRO CE 321R S2 Glass/Epoxy		
Time to Ds = 16	6 - 7 min.	1 - 2 min.
Time to Ds = 264	11 - 12 min.	2 - 4 min.
Maximum Density	600 - 700	600 - 700
SD/g	120 - 140	92 - 108
MTL #7 - ICI-Fiberite MXB 7701 S2 Glass/Epoxy		
Time to Ds = 16	2 - 3 min.	1 - 2 min.
Time to Ds = 264	4 - 5 min.	1 - 2 min.
Maximum Density	> 700	> 700
SD/g	> 135	> 113
MTL #8 - American Cyanamid CYCOM 5902 S2 Glass/Epoxy		
Time to Ds = 16	2 - 3 min.	1 - 2 min.
Time to Ds = 264	4 - 5 min.	2 - 3 min.
Maximum Density	400 - 630	600 - 740
SD/g	40 - 63	81 - 100

Notes: (1) Time to Ds = 16 is the time required to reach 75% light transmission. Time to Ds = 264 is the time required to reach 1% light transmission, (2) Test specimen surface temperature in smoldering mode is 350°C (662°F), (3) SD/G = smoke density/gram = Dm(corr)/unit mass of sample.

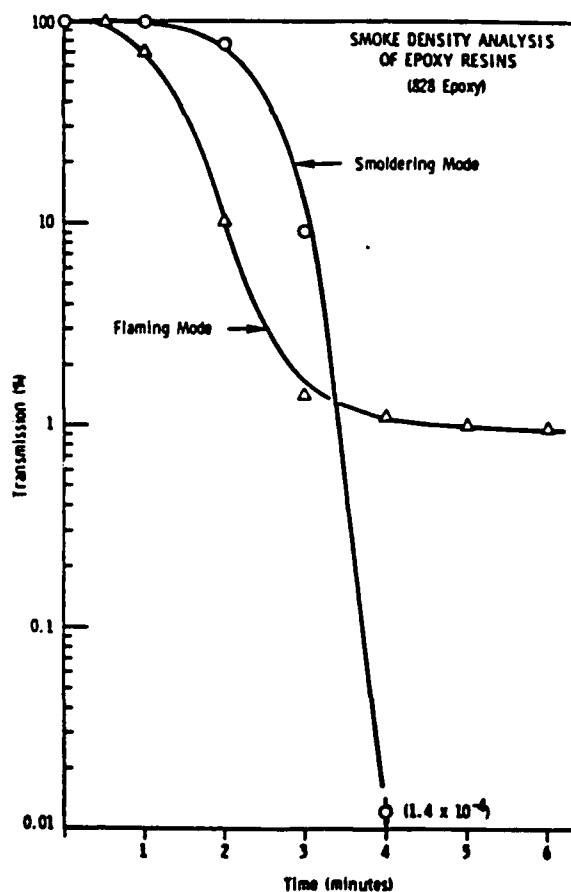


Figure 4. Light obscuration due to smoke.

Pyrolysis-Gas Chromatography/Mass Spectrometry (GC/MS)

The results of pyrolysis-GC/MS experiments performed as a means of assessment of pyrolysis effluent composition are shown in Tables 6 through 8 and Figures 5 through 7. Pyrolysis was performed at 900°C, in helium, because an oxidative atmosphere is not compatible with the analytical system at the present time. A total of 25 or 26 separated/identified constituents was obtained from each of the three composites. Many compounds appeared to be constituents of more than one resin formulation which is not unexpected.

Table 6. PYROLYSIS-GAS CHROMATOGRAPHY/MASS SPECTROMETRY
OF MTL #6, FERRO CE 321R; 900°C IN HELIUM

1.	Carbon Dioxide
2.	Propene
3.	Ethylene Oxide
4.	Bromomethane
5.	Propenal
6.	Acetone
7.	2-Butanone
8.	Water
9.	2-Propenyl ester of Acetic Acid
10.	Toluene
11.	Benzofuran
12.	Phenol
13.	Methylphenol
14.	Bromophenol
15.	Methylphenol
16.	Methylbenzofuran
17.	Dimethylphenol
18.	Ethylphenol
19.	Dimethylbenzofuran
20.	Isopropylbenzene
21.	Methoxystyrene
22.	Dibromophenol
23.	Bromo-t-butylbenzene
24.	Dichloroaniline
25.	Dichloroquinoline

Table 7. PYROLYSIS-GAS CHROMATOGRAPHY/MASS SPECTROMETRY OF
MTL #7, ICI-FIBERITE MXB-7701; 900°C IN HELIUM

1.	Carbon Dioxide
2.	Propene
3.	Ethylene Oxide
4.	1,3-Butadiene
5.	Bromoethane
6.	Propenal
7.	Acetone
8.	Water
9.	Propenol
10.	2-Propenyl ester of Acetic Acid
11.	2-Methyl-2-Propenoic Acid, Methyl ester
12.	Toluene
13.	Benzofuran
14.	Phenol
15.	Methylphenol
16.	Bromophenol
17.	Methylphenol
18.	Methylbenzofuran
19.	Ethylphenol
20.	Isopropylphenol
21.	Methoxystyrene
22.	Dichloroaniline
23.-24.	Dichloroquinoline
25.	Hydroxyphthalic Acid
26.	4-(1-Methyl-1-Phenylethyl)Phenol

Table 8. PYROLYSIS-GAS CHROMATOGRAPHY/MASS SPECTROMETRY OF MTL #8,
AM-CY CYCOM 5920; 900°C IN HELIUM

1.	Carbon Dioxide
2.	Propene
3.	1,3-Butadiene
4.	Bromoethane
5.	Propenal
6.	Acetone
7.	Water
8.	Benzene
9.	2-Propenyl ester of Acetic Acid
10.	Toluene
11.	Xylenes
12.	Styrene
13.	Isopropylbenzene
14.	1-Propenylbenzene
15.	Benzofuran
16.	Phenol
17.	Methylphenols
18.	Bromophenol
19.	Methylbenzofuran
20.	Ethylphenol
21.	Dimethylbenzofuran
22.	Isopropylphenol
23.	Methoxystyrene
24.	Dibromophenols
25.	Hydroxyphthalic Acid

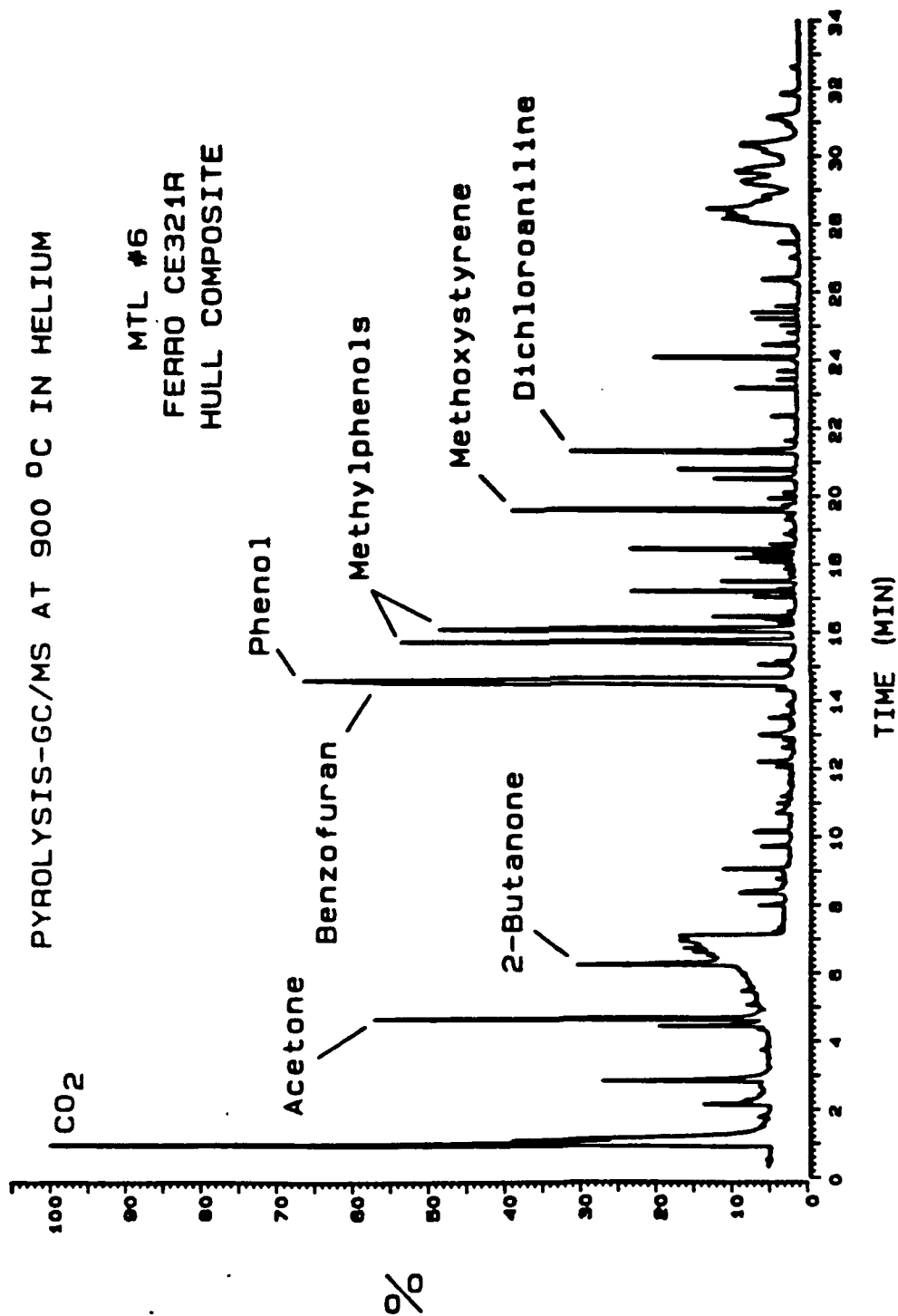


Figure 5. Pyrolysis-gas chromatography/mass spectrometry of epoxy-glass composite, MTL #6.

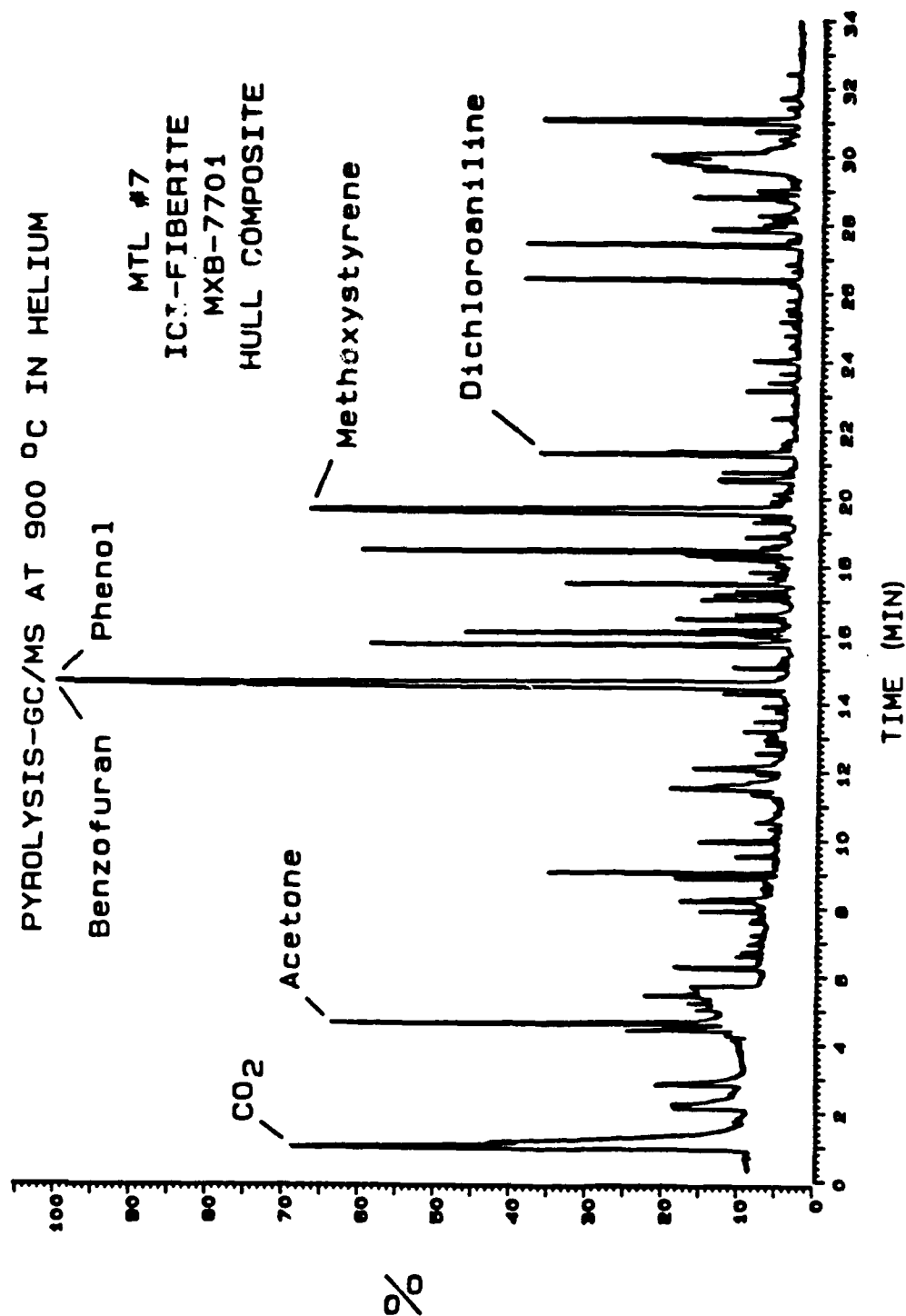


Figure 6. Pyrolysis-gas chromatography/mass spectrometry of epoxy-glass composite, MTL #7.

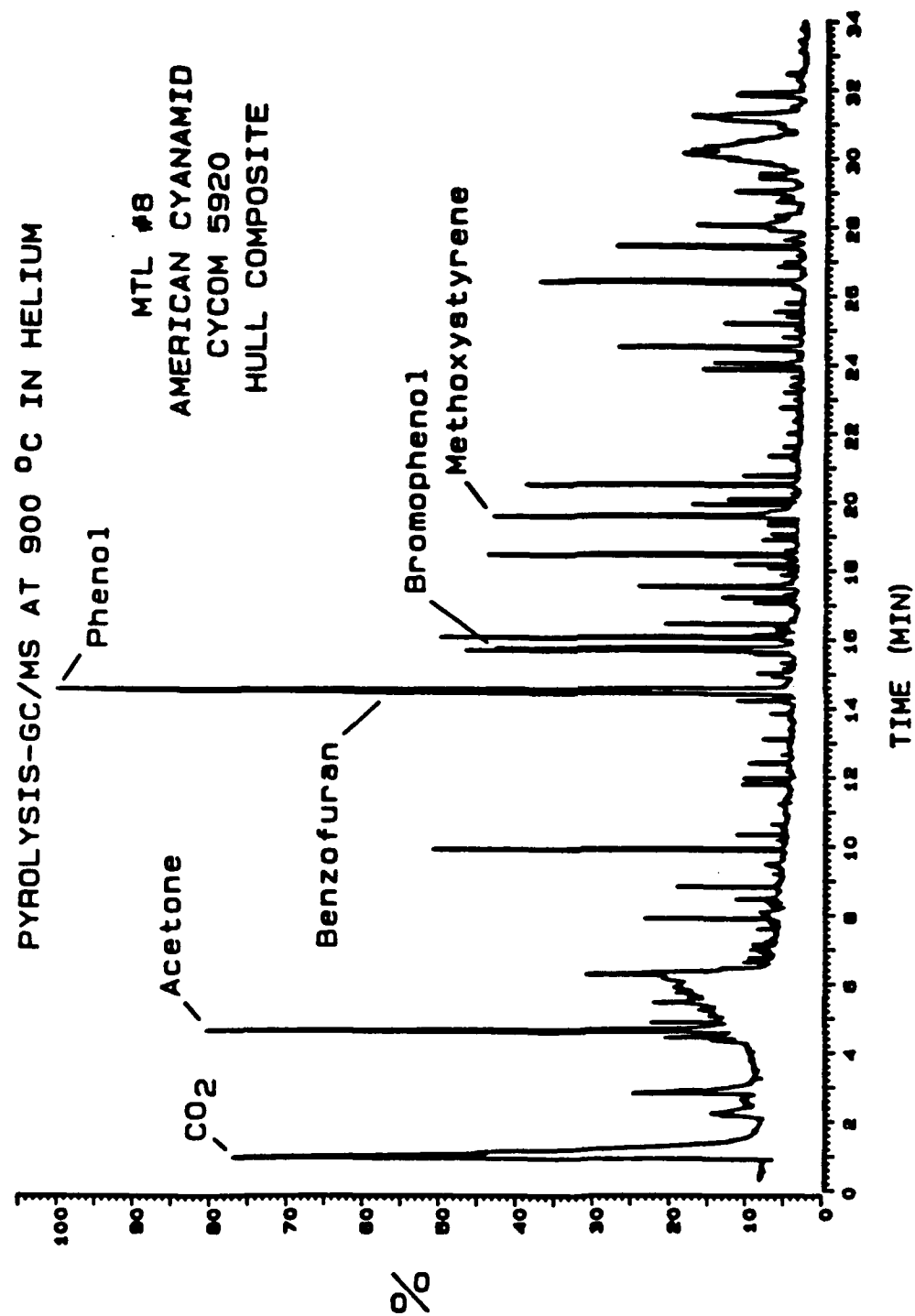


Figure 7. Pyrolysis-gas chromatography/mass spectrometry of epoxy-glass composite, MTL #8.

DISCUSSION

The results of the dynamic thermogravimetric analysis experiments, presented in Table 2 and Figure 1, demonstrate that composite samples MTL #6 through MTL #8 sustain little, if any, thermal damage below 200°C (392°F). The major mass loss occurs between 200°C and 800°C (392°F and 1472°F) due to decomposition of the matrix resin. In all compositions the maximum rates of mass loss occur at two temperatures; initially, 350°C (662°F) and again at approximately 525°C (977°F).

The results of the isothermal decomposition experiments, presented in Table 3 and Figure 2, illustrate the response of the composites when suddenly exposed to temperatures in the 300°C to 500°C (572°F to 932°F) region. Although most of the thermal damage to the composites occurs within the first five minutes at the higher temperatures, the results demonstrate that the material will withstand a temperature of 300°C (572°F) quite successfully for a longer period of time.

The results of the experimental determinations of OI and the temperature dependence of OI, as shown in Table 4 and Figure 3, indicates the degree of resistance to ignition and sustained combustion exhibited by MTL #6 through MTL #8. To keep the results in perspective, it should be remembered that normal atmosphere contains 21% oxygen. Therefore, any material whose OI is equal to, or less than, 21% would be expected to ignite and burn under normal atmospheric conditions. Materials whose OI is greater than 21%, but less than 26%, will ignite with more or less difficulty but would most probably self-extinguish upon removal of the flame source. Materials with oxygen indices greater than 27% would not be expected to ignite under normal conditions. Thus, it is not likely that any material examined in this evaluation would ignite under normal atmospheric conditions.

The importance of the temperature dependence data is realized when one considers that in a fire the thermal environment of a material will normally elevate due to the combustion of surrounding structures. The behavior of the material under examination, at elevated temperatures, then becomes important. In general, the oxygen requirement for sustained combustion will decrease as the temperature of a material increases; i.e., the OI decreases. When the OI falls to the level of oxygen present, at a given temperature, a flashover will occur and the material will combust. The overall fire load will be increased to the degree that new combustible material becomes involved.

If we consider the data shown in Table 4 at temperatures between 100°C and 300°C (212°F and 572°F) one can be certain that composite MTL #6 would sustain combustion at 300°C (572°F) and beyond. MTL #7 and #8 would have to reach a temperature in excess of 300°C (572°F) in order to sustain combustion since their OI values are somewhat higher at that temperature level. The increase in OI of composites MTL #6 to #8 in the 100°C (212°F) region is behavior similar to that noted earlier⁶ with other polymers.

The smoke generation characteristics of MTL #6 to #8 are presented in Table 5 and a representative data plot is shown in Figure 4. Data for time to Ds = 16 indicates the amount of time available before it would be difficult to locate an escape route on the order of 10 feet away. Time to Ds = 264 indicates the time before a light transmission level is reached where vision is no longer possible. The value of maximum optical density (Dm) obtained can be used as a general indicator of the smoke generation classification of the material under evaluation. The order of magnitude values would be:

6. MACAIONE, D. P. Flammability Characteristics of Some Epoxy Resins and Composites. U.S. Army Materials Technology Laboratory, AMMRC TR 83-53, September 1983.

- Low Smoke Generation --- $D_m = < 200$
- Moderate Smoke Generation --- $D_m = 200 - 450$
- High Smoke Generation --- $D_m = > 450$

The exact numerical values may be academic once a value of $D_m = 264$ is exceeded; however, the maximum SD does indicate the relative smoke load produced by each material.

Perhaps a more instructive parameter is the value of SD per gram (SD/G) because it provides a direct relationship between a quantity of material and the level of smoke generation. Of the materials evaluated in this study, MTL #8 produced the lowest value of SD/G. The value of time to $D_s = 264$ was, at least in smoldering mode, shorter by a factor of two for MTL #8 when compared to MTL #6. Under these conditions, valuable *escape time* would be lost during a fire.

Attempts to evaluate the toxicity of combustion effluent from burning organic materials have resulted in an ongoing debate within the fire science community. Apart from the fact that the thermal environment of a fire produces a complex set of reaction conditions that may seldom be duplicated in any two successive events, the combustion of organic material will always produce carbon monoxide and carbon dioxide, in large quantity, in addition to all of the other species produced. For these reasons we have elected to take an instrumental approach to evaluating the potential toxicity of combustion effluent generated by organic materials. The experimental results obtained can then be reviewed for the presence of particularly hazardous species.

Within this context, the data presented in Tables 6 to 8 and Figures 5 to 7 indicate that the components of the effluent resulting from the pyrolysis of MTL #6 to #8 are quite similar. In fact, of the 25 or 26 compounds detected in the pyrolysis effluent of these composites, nearly half of them are found in all three specimens. No halogen acids (HCl or HBr), nor HCN, were detected in the effluent under our reaction conditions.

CONCLUSIONS

The fire survivability of a U.S. Army combat vehicle and crew has been a major concern and obstacle to the general application of structural composites by the military. An understanding of the flammability behavior and overall fire tolerance of organic materials is crucial to the proper selection of materials which must occur in the initial stage of vehicle design.

The study described in this report was undertaken to assess the flammability characteristics of fiber-reinforced epoxy composite materials in view of their potential application in combat vehicle systems. Three fiber-reinforced composite compositions were evaluated. Considering the potential hazards due to fire and the generation of heat and combustion products the results indicate that these would most probably be limited to the ignition zone. The performance of the composites evaluated, although acceptable, could be enhanced by co-curing an outer layer of phenolic composite; e.g., MTL #5, to the basic epoxy material which would function as a structural core.

Based upon the results obtained in this investigation it has been shown that a military vehicle fabricated from these fiber-reinforced composite materials would not represent an unusual fire hazard solely by virtue of its construction and that composites such as the ones examined in the current study would most likely respond in such a manner as to increase the fire survivability of the system.

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U.S. Army Materials Technology Laboratory
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FLAMMABILITY CHARACTERISTICS OF FIBER-
REINFORCED EPOXY COMPOSITES FOR COMBAT
VEHICLE APPLICATIONS -
Domenic P. Macalione

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Key Words

Composite materials
Fire resistance
Polymers

Technical Report MTL TR 92-58, August 1992, 18 pp-
illus-tables, D/A Project: 16263102D071

The use of composites in U.S. Army systems as a means of decreasing weight and enhancing survivability, without reducing personnel safety, has been considered for some time. The U.S. Army Materials Technology Laboratory (MTL) successfully demonstrated in an earlier program that a ground vehicle turret could be fabricated from fiber-reinforced composite material. This technology was successfully extended to the fabrication of a composite vehicle hull in an earlier phase of the current program. Organic polymers are one of the major constituents of fiber-reinforced composites. As components of military systems these materials are expected to survive combustion and pyrolysis processes associated with fires. It is, therefore, necessary to develop an understanding of the flammability behavior of composite materials in the early design stages of a military vehicle such as the Composite Infantry Fighting Vehicle (CIFV), the Advanced Systems Modification (ASM), or any future U.S. Army combat vehicle. The present study attempts to characterize the flammability behavior of composite materials associated with Phase III of the CIFV Hull Program in terms of accepted fire-resistant material evaluation parameters.

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